

PROJECT TITLE : ANALYTICAL INVESTIGATIONS  
PERIOD COVERED : MAY 18 - JUNE 17, 1981  
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#### ADSORBENTS

The main physical parameters of the charcoal (type: mineral charcoal) used for cigarette Helikon (Hungaria) were determined (1) : Specific surface  $215 \text{ m}^2/\text{g}$ , pore volume  $0.37 \text{ cm}^3/\text{g}$ , pore radius  $19.4 \pm 1.0 \text{ \AA}$ , mesopore volume  $0.16 \text{ cm}^3/\text{g}$ , micropore volume  $0.21 \text{ cm}^3/\text{g}$ .

The adsorbent Diatomite (manufacturer : Wüthrich-Yverdon, Switzerland), previously studied in powder form (2), was reinvestigated in particulate form (diameter 1-3 mm) (3). The organic gas-phase, NO and CO are not retained by the adsorbent. 15% retention of the organic gas-phase and no retention of NO and CO was obtained after breaking the particulates into small particles of 0.5-1 mm granulometry. For taste evaluation several cigarettes with Diatomite in the filter were prepared and are presently being tested.

#### CF IN COMPETITIVE BRANDS

##### West Germany

With regard to the special permissions for CF recently granted to BAT and BRI in Germany, the main brands of BAT (HB, Lord Extra, Krone, Kurmark, Kim, Pall Mall NF, Benson & Hedges, Kent de Lux, Prince Denmark) and BRI (Lux, Peer 100, Windsor de Lux, Dunhill International) manufactured during the period March 1981 through May 1981, were analysed for CF. None of the brands contained CF (4).

##### Switzerland

Caballero F and NF, two new brands, both manufactured in the Netherlands by Laurens-Rothmans, were found to contain 11 respectively 14 ppm of CF (5).

## TRIACETIN

Two triacetin samples submitted by QC (6) were extensively investigated by applying different analytical techniques.

Sample A : Triacetin used in Bergen op Zoom (ex Eastman). MLF produced with this triacetin were not accepted by panel decision.

Sample B : Triacetin from FTR was sent to Bergen op Zoom for tests. Cigarettes were considered acceptable. The origin of both triacetin samples was the same i.e., both are from the same Eastman central storage tank in Rotterdam.

(GC)<sup>2</sup> : Chromatograms of A and B are absolutely identical.

Pyrolysis GC : Pyrolysis of the samples at 200 to 600°C/20 msec. (GC Packard 427, column 1.5 m x 2 mm, OV 17 (5%), isotherm 220°C) did not show any difference between the samples.

HPLC : Chromatograms obtained from both samples on RP 8 (25 cm/10 µ) with CH<sub>3</sub>CN 27% / water 73% at 35°C are identical. No high-boiling oligomeric materials were detected.

TLC : Investigations on normal and reverse phase silica gel using a variety of solvent systems and visualizing agents failed to reveal any difference between the two samples.

pH : Measurements of the aqueous extract of triacetin did not reveal any free acidity in either sample.

Metals : The solutions were analysed by plasma emission spectrometry. Seven parameters were selected for quantitative measurement. Results are as follows (in mg/l):

	Na	Zn	Pb	Fe	Ni	Cr	Sn
Sample A	34	1,8	1,1	1,6	0,3	0,2	17
Sample B	45	1,2	0,7	4,1	0,3	0,7	17

The differences between the samples do not appear significant.

Refractive Index (22°C) : Sample A 1.43425 ; Sample B 1.43419  
The difference between the samples is insignificant.

Karl-Fischer : The water content of Sample B (0.17%) is slightly higher than that of Sample A (0.10%).

IR Spectroscopy : The IR spectra of Samples A and B are identical between 500 and 3500  $\text{cm}^{-1}$  but show for sample B a more intensive v O-H band at 3600  $\text{cm}^{-1}$  (higher water content.)

Thermochemical Methods : Differential scanning calorimetry failed to reveal any differences between the two samples. On cooling the samples the glass transition point was identical in both cases. The samples did not crystallize on further cooling ( $-70^{\circ}\text{C}$ ), but remained in the glassy state. This prevented melting point determinations.

Results of LC- (using infrared or refractive index detection) and headspace analysis are still pending.

#### N-NITROSAMINES

Four brands from UK, Rothmans KS, John Player Special, Benson & Hedges and Embassy No. 1 KS, were analysed for DMN and NPY. Values found are between 147 and 225 ng/cig. for DMN and 40 to 66 ng/cig. for NPY (7).

NNN, NATB and NNK, as well as DMN and NPY, were analysed in 12 tobacco extract samples and in 7 additives from Project LEAR (8). Amounts of tobacco specific N-nitrosamines found in extracts are within the ppb-range. DMN and NPY were not detected (9).

#### WS-14

The determination of the Threshold Odor Value in sidestream smoke of WS - 14 has been requested by R.M. Ikeda, PM Richmond.

The concept of simultaneously smoking both control and test cigarettes in closed hoods has been discussed with Ikeda. A test panel should evaluate the smoke samples with simultaneous GC analysis of WS - 14 to determine the threshold odor value.

The modifications of existing equipment required to carry out this work are relatively minor and have been requested from the workshop.

## OTHERS

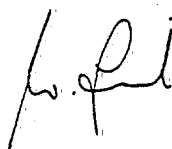
Routine analyses were performed on potassium, calcium, magnesium, phosphate and sulfate in 29 samples submitted by Biotechnology.

## REPORT WRITTEN

Genoud-Y. "Triacetin and Triethylene Glycol Diacetate in Filter Plugs by Capillary Gas Chromatography" Analytical Method, June 1981.

## REFERENCES

- (1) Verbal request from Pittet.-M. to Genoud.-Y.
- (2) Genoud.-Y. PME Monthly Progress Report, April 1981
- (3) Memo from Krasna.-B. to Genoud.-Y., March 23, 1981
- (4) Memo from Lecoultre.-E. to Singer.-Z., June 18, 1981
- (5) Memo from Lecoultre.-E. to Nagel.-P., June 18, 1981
- (6) Memo from Widmer.-A. to Fink.-W., May 13, 1981
- (7) Memo from Fink.-W. to Gaisch.-H., June 19, 1981
- (8) Request from Ruf.-C to Moser.-F., May 25, 1981
- (9) Memo from Fink.-W. to Ruf.-C., June 22, 1981



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Fink-W.